

Advanced Materials Characterization at the High Temperature Materials Laboratory

**Arvid Pasto
Director,
High Temperature Materials Laboratory
Oak Ridge National Laboratory
April 2002**

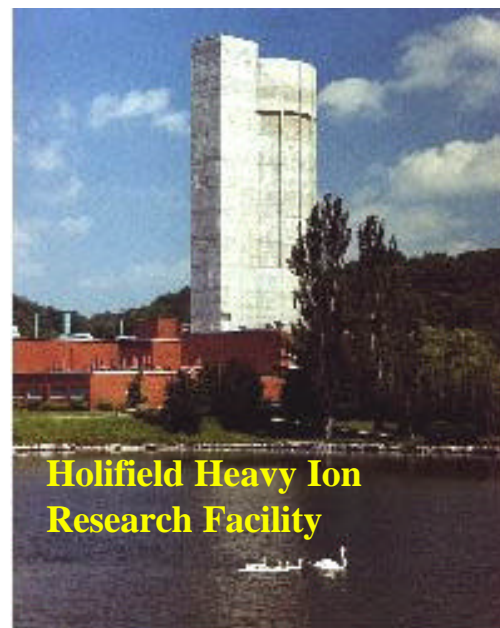
Department of Energy Labs Are Good Partners for R&D

- Most of the US Government's materials-related research and development is done by DOE
- DOE National Labs perform R&D in all conceivable areas of advanced materials
 - structural
 - electronic
 - magnetic
 - optical
 - biological
 - etc.

Oak Ridge
Argonne
Brookhaven
Los Alamos
Sandia
Idaho
Berkeley
Livermore

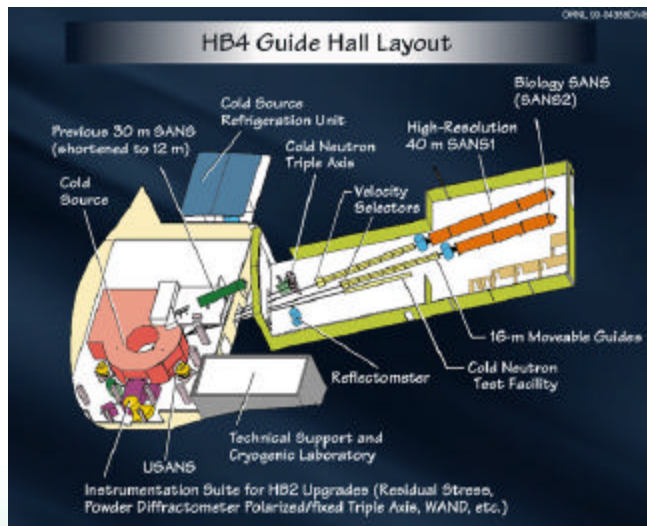
Many Mechanisms Exist for Working With DOE Labs

- **Contracts from the labs to industry, academia**
- **CRADAs**
- **Work for Others**
- **Collaborative efforts**
- **Fellowships**
- **Personnel exchanges**
- **User facilities**



User Facilities Provide Access to Special Equipment and Expertise

- Most laboratories have one or more
- These labs contain equipment and skilled staff that no private company or university could afford



- synchrotrons (light sources)
- reactors (neutron beams)
- accelerators (particle beams)
- high power electron optics
- etc.

User Program Requirements

- (1) User agreement between your institution and ORNL (legal, IP items)**
- (2) Technical proposal describing your project**
- (3) Clearance into the laboratory**
- (4) Funds into ORNL (if required)**
- (5) You come do the work here with us**

(1) thru (3) can be accomplished simultaneously; schedule is typically dependent on your lawyers or our clearance process (foreign nationals from sensitive countries take ten weeks minimum). Work cannot begin without (4).

Oak Ridge National Laboratory Alone Has Seventeen User Facilities

- Metals Processing Laboratory User Center (MPLUS)
- Shared Research Equipment Program (ShaRE)
- Buildings Technology Center
- *High Temperature Materials Laboratory*
- *NTRC*
- Numerous others

The HTML is a DOE-Designated User Facility at Oak Ridge National Lab

Arvid Pasto, Director (865-574-5123)
Billie Russell, Administrative Assist.
Jesse Whittenbarger



Six HTML user centers focused
on materials characterization

www.ms.ornl.gov/htmlhome

Materials Analysis
Thermophysical Properties
Diffraction

Residual Stress
Machining, Inspection, and Tribology Research
Mechanical Characterization and Analysis

The HTML Programs are funded by DOE's Office of Transportation Technologies
Dr. Sidney Diamond, DOE Program Manager

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HTML Capabilities are Available to Users for Hands-On Materials Characterization Research

Materials Analysis



**Characterize
structure and
composition**

Mechanical Characterization and Analysis



**Characterize mechanical
properties and performance**

Thermophysical Properties



**Measure physical
properties as a function of
temperature**

HTML Capabilities are Available to Users for Hands-On Materials Characterization Research

Diffraction



**In-situ characterization
of crystalline materials
(x-ray and neutron diff.)**

Residual Stress



**Characterize and map
residual stress and
texture**

Machining, Inspection and Tribology



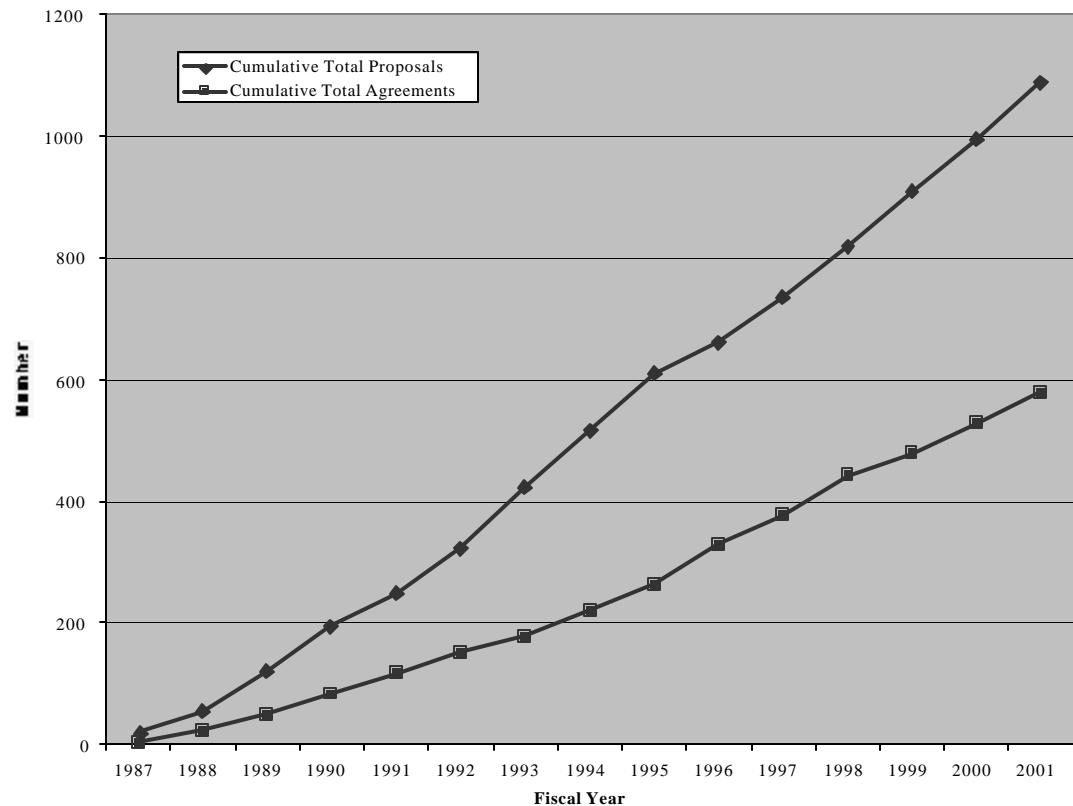
**Investigate grinding
processes and
characterize surfaces**

HTML Programs and Staff Have Benefited Industry and Universities

- Many institutions have signed User Agreements:
353 industries
205 universities (thru FY2001)
21 government agencies
579 total institutions
- These have resulted in over 1080 projects
- Industries range from small (7 people) to large (GM, Ford, GE, Honeywell,.....)
- Many repeat visits from user institutions indicates success of projects

HTML User Statistics Show Continued User Agreement, Proposal Growth

- **Agreements up (thru FY01, to 579)**
- **Proposals up (thru FY01, to 1084)**

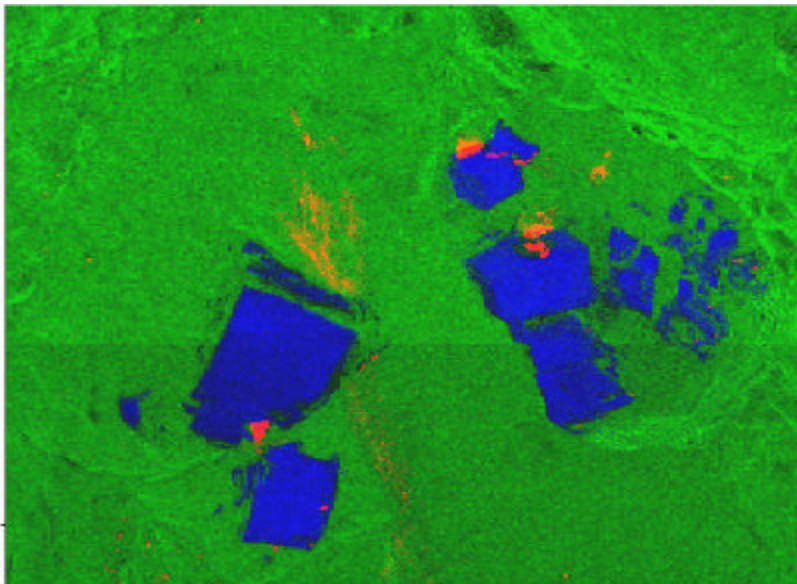


Some FY 2001 Projects Have Been Especially Noteworthy: Materials Analysis

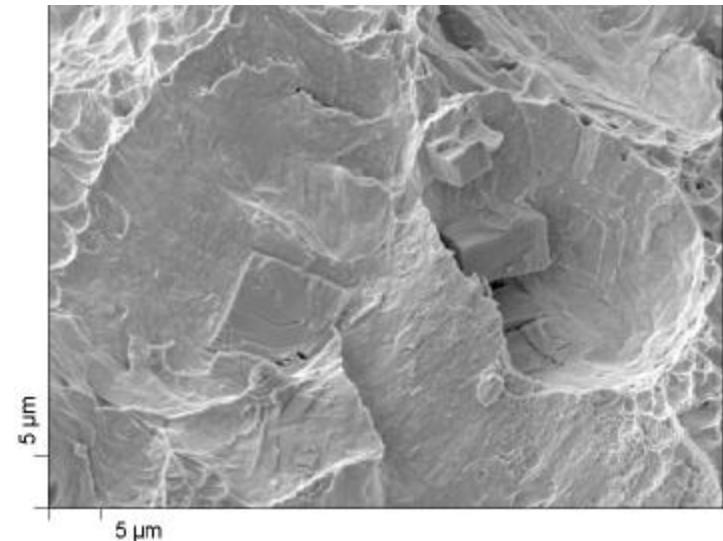
- **Zeolite molecular sieves**
- **Diesel engine exhaust particulates (SwRI)**
- **Chromium depletion in high-temperature alloys**
- **Single-wall carbon nanotubes**
- **Thermal barrier coating “template” layers**
- **Laser-treated surfaces(UTSI)**
- **Exhaust emission catalyst (CATERPILLAR)**

Harry Meyer and Cynthia Hsieh, Caterpillar Studied Element Distribution on Fractures

The secondary electron image shows the fracture surface of high-strength steel sample. Note the presence of “cuboid” structures at the center of the fracture pits. The cuboids are TiN as determined by Auger analysis.



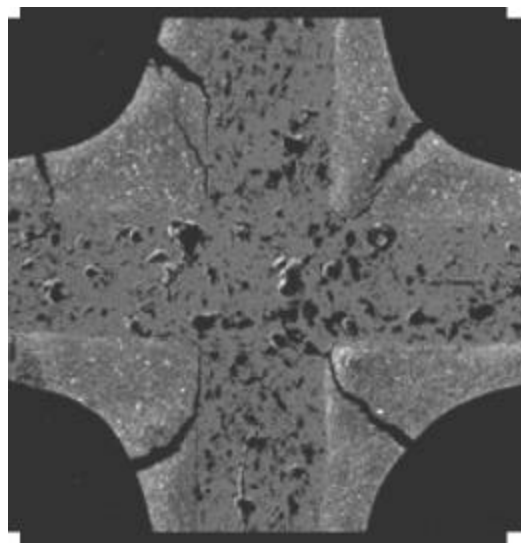
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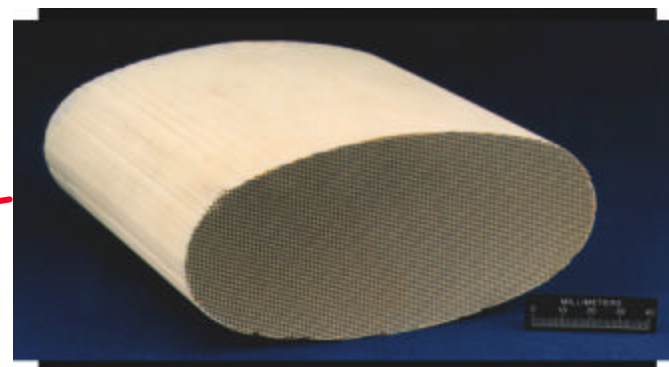
Elemental maps of **iron**, **titanium**, and **sulfur** reveal the presence of sulfur at the interface between the steel matrix and the cuboids.


UT-BATTELLE

The HTML is a National Leader in Characterizing Catalyst Materials



Automotive catalyst brick

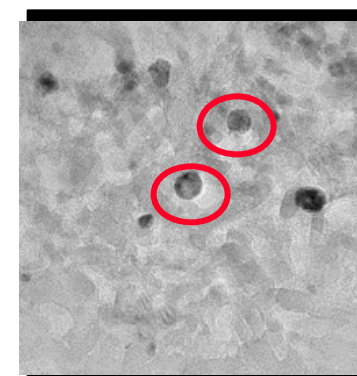
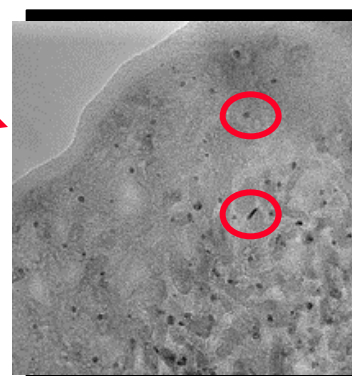


SEM shows cordierite substrate with washcoat in corners

HTML has/had catalyst activities with

AC Delphi
U. Delaware
U. New Mexico
Ford Research Laboratory
Cummins Engine Co.

W.R. Grace
General Motors
United Emission Catalyst
AlliedSignal
U. Michigan



High-resolution TEM of catalyst particles shows changes

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HTML Placed ACEM Contract 9/22/00; Instrument due at ORNL Summer 2003

ACEM will:

- be first probe corrected STEM/TEM in US
- have sub-Å resolution for structure
- have sub-Å resolution for chemical analysis
- resolve single Pt atoms on alumina
- be totally automated and remote ready
- very user friendly
- be delivered spring/summer 2003
- require a special facility- low EM fields, etc.

JEOL will provide us an interim STEM/TEM

- with 1.4 Å structural and chemical resolution
(much better than we have now, and excellent for catalysts)
- worth about \$1.4 M
- to be delivered within a year



New Building Will Be Styled to Mesh With HTML



**Currently called the
Advanced Materials
Characterization
Laboratory (AMCL)**



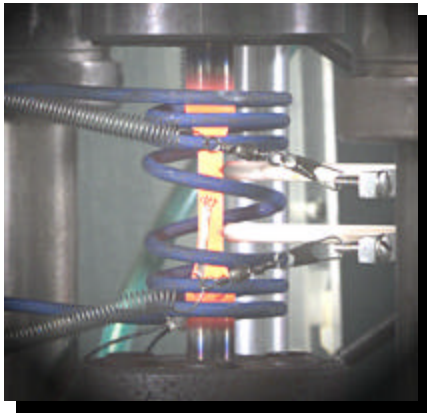
Mechanical Characterization and Analysis User Center has Complete Facilities

- **28 Electromechanical universal testing machines**
- **23 servohydraulic universal testing machines, at rates up to 1000 Hz**
- **14 test frames with pneumatic actuators**
- **166 dead-weight frames**
- **3 Biaxial (axial/torsional) test frames**
- **Test facility for internal pressurization of tubular components**
- **30 Environmental & High Vacuum chambers**
- **2 Nanoindenters**
- **1 Micromechanical test unit**
- **1 Thermal shock facility**
- **1 Infrared camera**

Some FY 2001 Projects Have Been Especially Noteworthy: Mechanical Characterization

- **Residual stresses of ceramic composites (WESTINGHOUSE)**
- **Creep of magnesium alloys (NORANDA) and
of nanocomposites (UC-D)**
- **Low cost silicon carbide (POCO)**
- **Zirconia-based ceramics (CATERPILLAR)**
- **Metallic foams (PORVAIR) and ceramic foams (ULTRAMET)**
- **Nd-Fe-B magnets (Magnequench/NCSU)**

Development of Low-Cost Austenitic Stainless Steel for Gas-Turbine and Diesel Engine Components



- Diesel portion funded by OTT HVPM program
- CN12 (Fe-25Cr-13NiNbCN alloy) for exhaust manifolds and turbocharger housings.
- Comparison with SiMo ductile cast iron and the baseline CN12
 - strength, isothermal fatigue, and creep.
- Alloy development and screening at ORNL
 - "super" CN12 with 10 times the creep resistance at 850°C
 - "super" CF8C (Fe-20Cr-12NiNbC alloy)
- Alloys can be used at 850°C, are comparable to standard CN12 but cheaper!
- Invention disclosure on the modified CN12 and modified CF8C stainless steels

Mechanical Properties of Aluminum-Intensive Vehicles

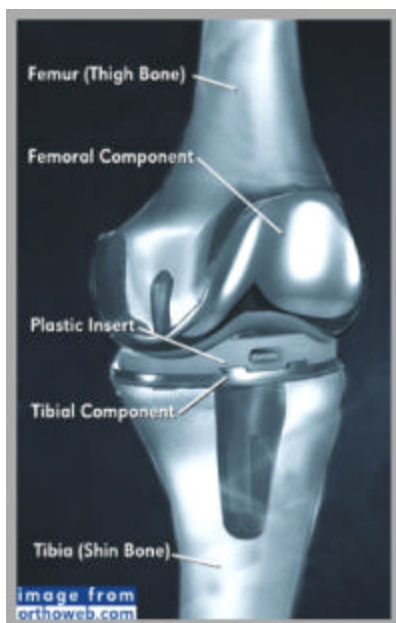


- Automobiles that triple the efficiency of today's cars without sacrificing performance, utility, cost of ownership, or safety,
- Weight reductions of 40%.
- Lightweight metals, plastics, and composites.
- Computational models to simulate the response of automotive structures in collisions (NHTSA-DOE)
- Aluminum intensive vehicle (Audi A8).
- Properties in uniaxial tension, compression and shear to generate the required data for modeling purposes.
- motor mounts, shock absorbers, and body panels.

Nanowear in Knee Replacements



Professor Martine LaBerge
Sunita Ho

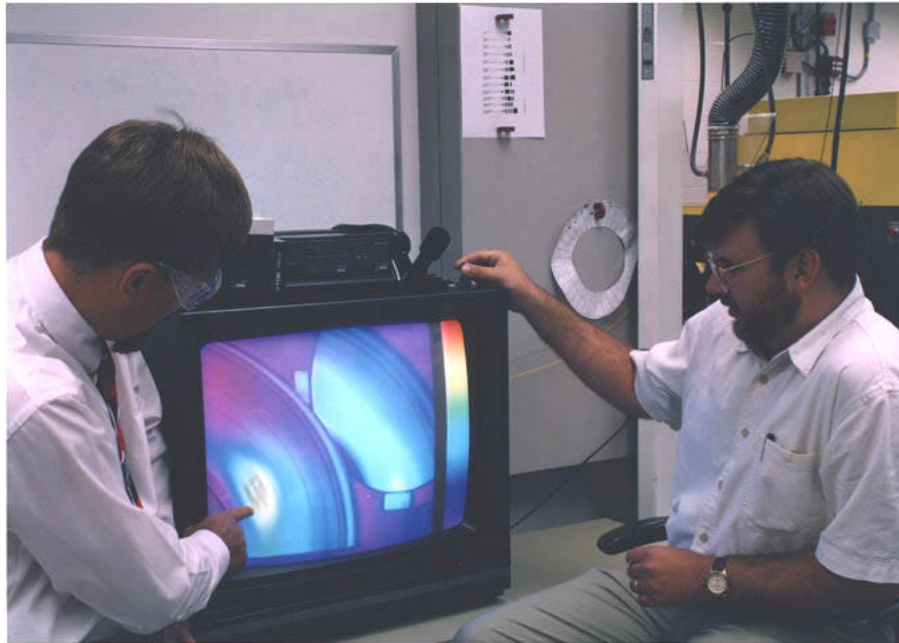


- Total knee joint replacement (TJR)
- CoCr alloy component articulating against ultra high molecular weight polyethylene (UHMWPE).
- Production of wear particles. Wear debris produced with polyethylene components has shown to induce osteolysis
- Understanding of polymeric wear in TJRs
- Quantification of nanomechanical properties of polymeric bearing material as a function of the nanotopography
- Elastic modulus : 2.29 ± 0.1 GPa for the elastic modulus along the surface of the compression molded tibial UHMWPE insert
- Hardness values of 0.07 ± 0.01 GPa

Some FY 2001 Projects Have Been Especially Noteworthy: Thermophysical

- **Thermal conductivity/diffusivity of:**
linear cellular materials(GT)
single-wall nanotubes in coatings (Rice Univ.)
TBC's (Siemens-Westinghouse)
- **High speed IR camera studies of:**
ball grid array inspection technique (Motorola)
GaN heterostructure FET temperature distrib. (USC)
SiC/SiC composite tensile test heating (UTK)
aircraft carbon brakes (SIU)
- **Thermal analysis of:**
“building block” mixed metal oxides (UTK)

High Speed IR Camera Used at Ford, GM, Bosch to Image “Hot Spots” on Brake Rotors

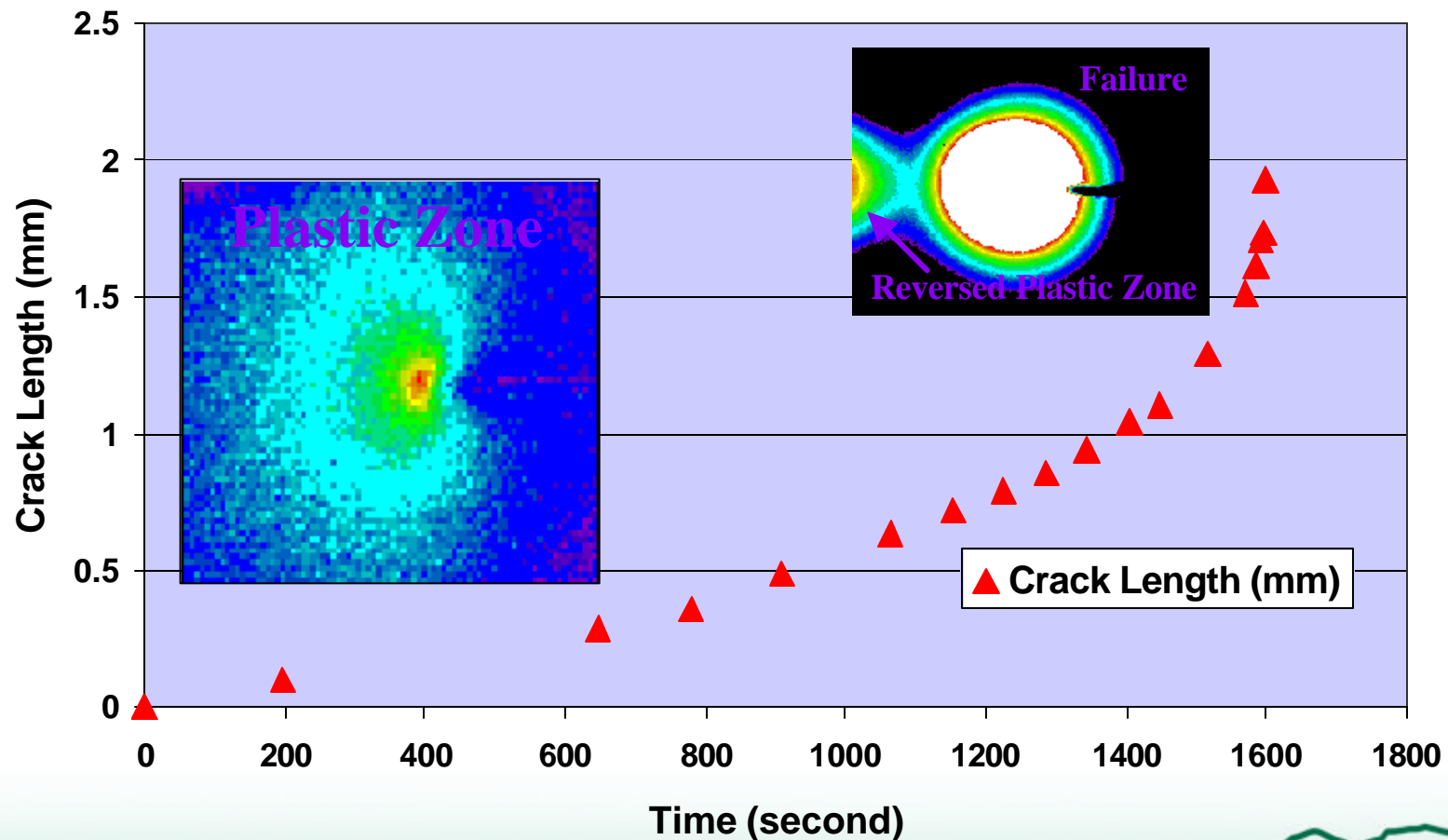


Ford engineer (left) and HTML scientist view TV monitor showing “hot spots” on spinning brake rotor under heavy braking

These thermoelastic instabilities had long been predicted but never before imaged directly

Application of IR Imaging to Monitor Superalloy Fatigue Crack Growth

TPUC User Project with the University of Tennessee



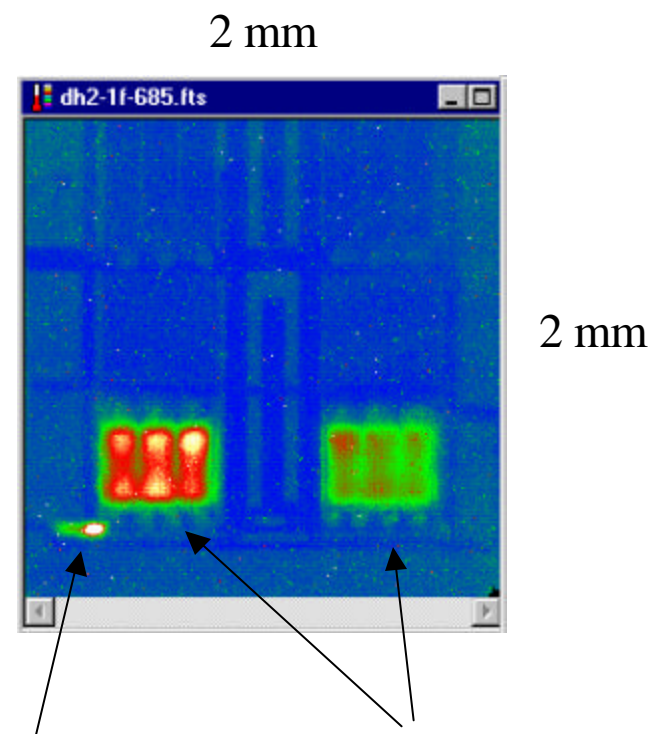
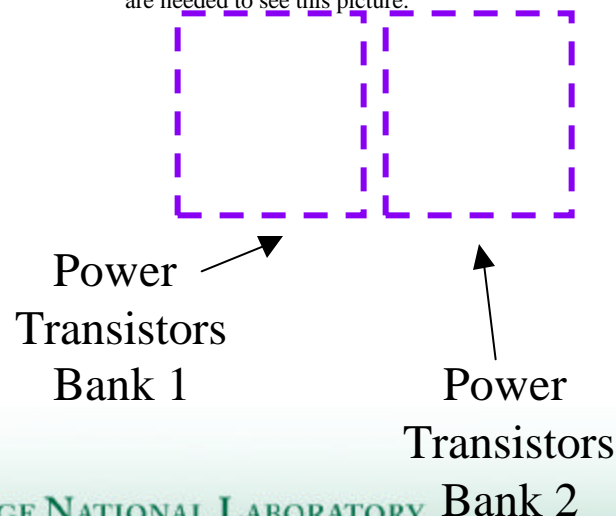
A Microscope Lens Allows the Study of Integrated Circuit Power Transistors During Operation and Failure

TPUC User Project with Motorola

Movie During Failure

Click on movie to play

QuickTime™ and a
Cinepak decompressor
are needed to see this picture.

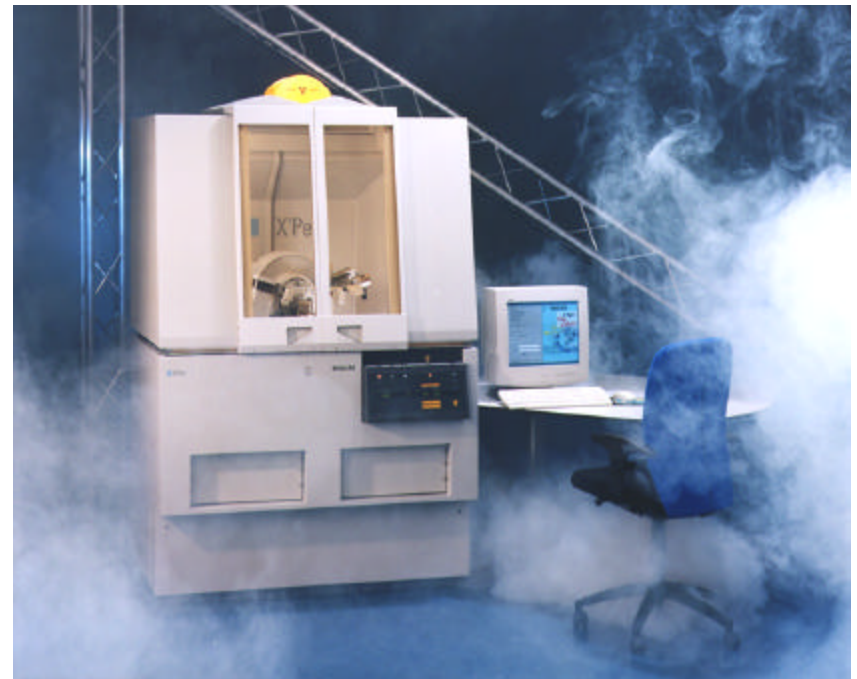


Some FY 2001 Projects Have Been Especially Noteworthy: Diffraction

- **Phase transitions in the $\text{NH}_4\text{NO}_3\text{-KNO}_3$ solid solution - U. of Nevada, Reno and TRW, Inc.**
- **Structure of substituted hexagonal barium aluminum silicate by neutron diffraction - Corning**
- **Negative thermal expansion materials - Georgia Tech**
- **In-situ studies of Zr_2Fe hydrides - U. of Nevada, Reno**
- **Nitridation of two-phase Cr-Pt alloys - University of Tennessee**
- **Development of nanocrystalline ceramic particles and thin films - ORNL-BES**
- **Thermal expansion anisotropy in ternary molybdenum silicide intermetallics - ORNL-BES)**

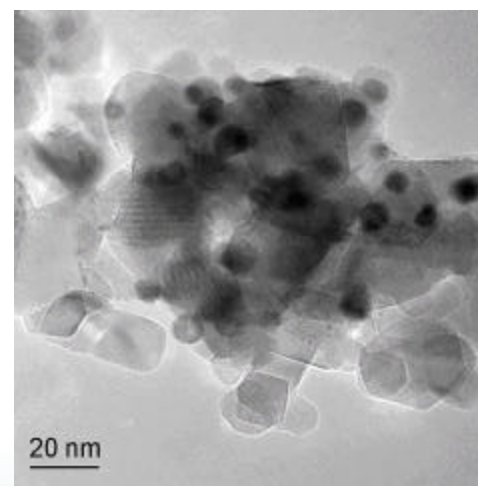
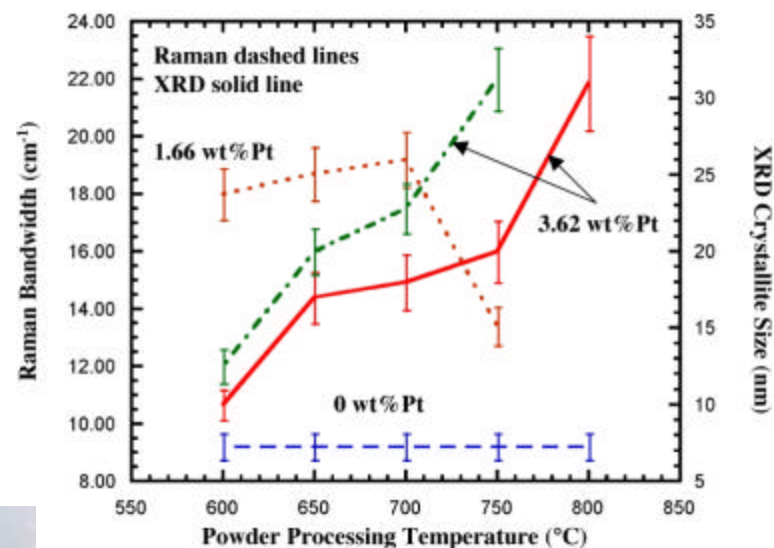
Philips Has Consigned an XRD System to DUC

- State-of-the-art machine, with elevated temperature capability
- Instrument worth >\$345,000
- Installed November 2000
- Residence time 12-18 months
- HTML investing in chiller, lab preparation, etc.



Cummins Characterized New Catalyst

Goal: To meet 2007 emission requirements
Catalytic activity decreases with surface area
 ⊢ **Platinum (Pt) particle size increases**
X-ray diffraction, Raman & TEM
 techniques track Pt size change
Collaboration: HTML/ORNL &
 Cummins Engine

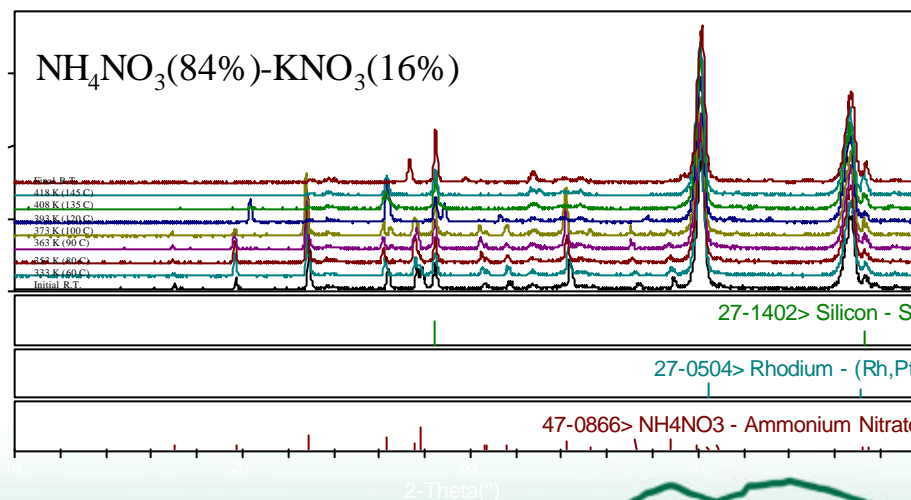


In Situ X-Ray Diffraction Helps Researchers Understand Automotive Components at Temperature

Ammonium nitrates are oxidizers in new generation automobile air-bag propellant and need be phase stable between -50 °C to 150 °C



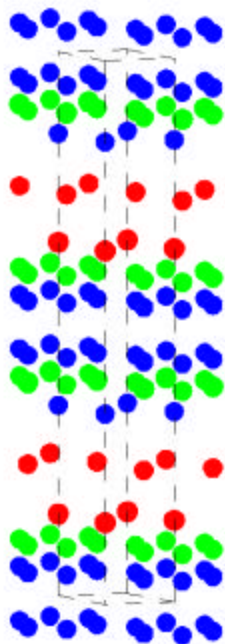
TRW Vehicle Safety Systems



High Temperature Neutron Diffraction Resolved Correct Structure for Ti_4AlN_3



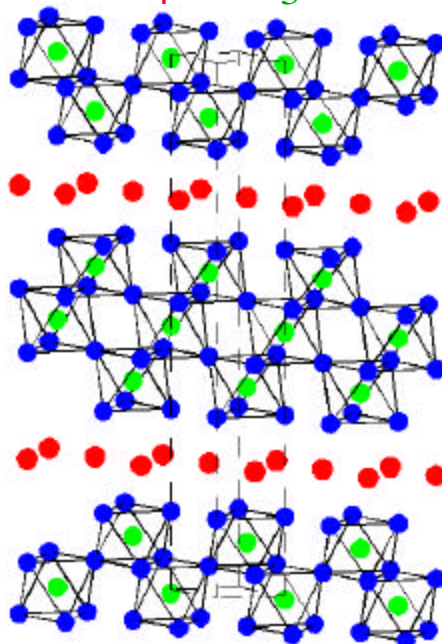
Structure
originally
proposed
by Schuster
and Bauer



$$a = 2.9875,$$

$$c = 23.350 \text{ \AA}$$

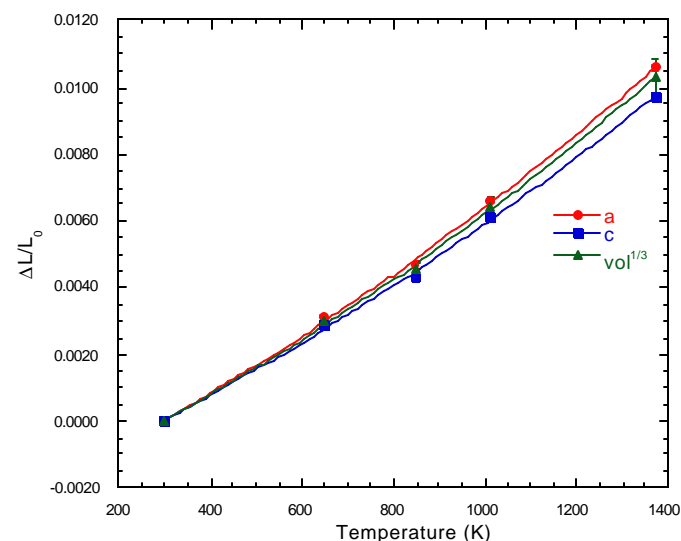
$$P31c$$



$$a = 2.9888(3)$$

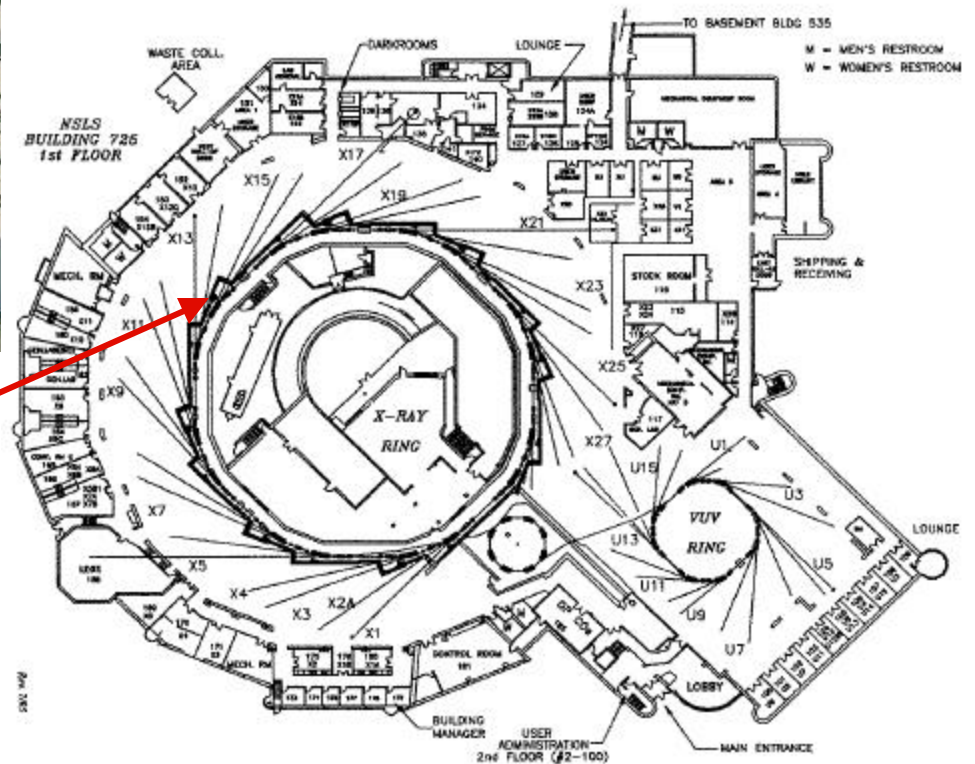
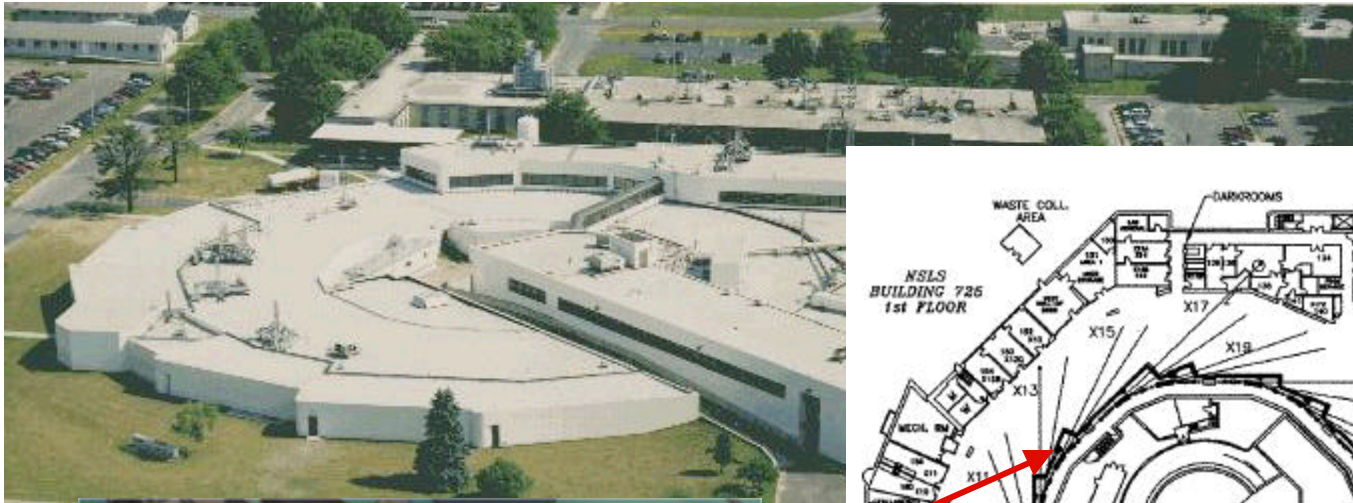
$$c = 23.382(1) \text{ \AA}$$

$$P6_3/mmc$$



Michel Barsoum
Tamer El-Raghy
Dept. of Materials Engineering
Drexel University

HTML Has Taken Over Operation of the X14A Beamline at the NSLS



Capillary Furnace
On HTML's
Beamline

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X14A: Proven Value to OTT-OHVT Customers

<u>Customer</u>	<u>Project</u>
• Cummins Engine	Stresses in surface treated steels via high energy x-rays
• Norton	Induced stress from innovative grinding wheels
• Corning	Thin films for video display
• Tycom	WC drill bits
• Quantum	Magneto-resistive heads
• GE/ATS	HT residual stress in TBCs
• Rutgers Univ.	Electroactive ceramics
• Univ. of Pitts.	Growth stresses in protective oxide coatings
• Drexel Univ.	Ductile ceramics for HT applications
• Univ. of Conn.	Phase ID and Residual Stress in TBC
• S. Illinois Univ	Surface layers on brake pads after friction testing
• Georgia Tech	Cation ordering in magnetic nanoparticles

>100 User days in FY99 and also in FY00

Some FY 2001 Projects Have Been Especially Noteworthy: Residual Stress

- **Residual stress in directionally solidified eutectics (ORNL)**
- **Residual stress in aluminum engine blocks (Honda)**
- **Residual stress in laser-bonded coatings (UTSI)**
- **Friction stir welds in aluminum (USC)**
- **Completed large specimen x-ray stress-mapping facility (ORNL)**

Residual Stresses Measured in Al Engine Blocks



Machining releases constraints allowing pre-existing residual stresses to cause distortions

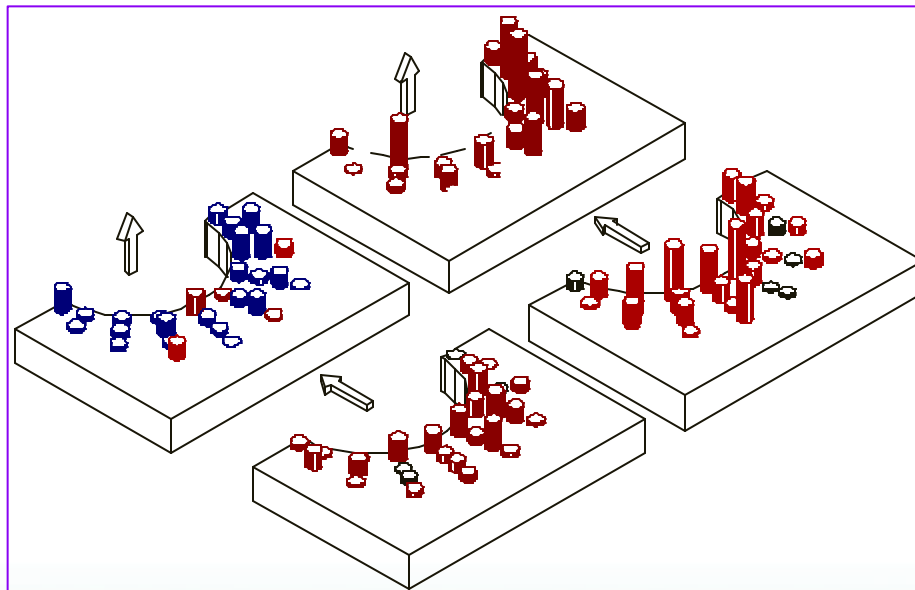
Requires additional heat treatments prior to machining

Thus, a major production-volume limiting step!

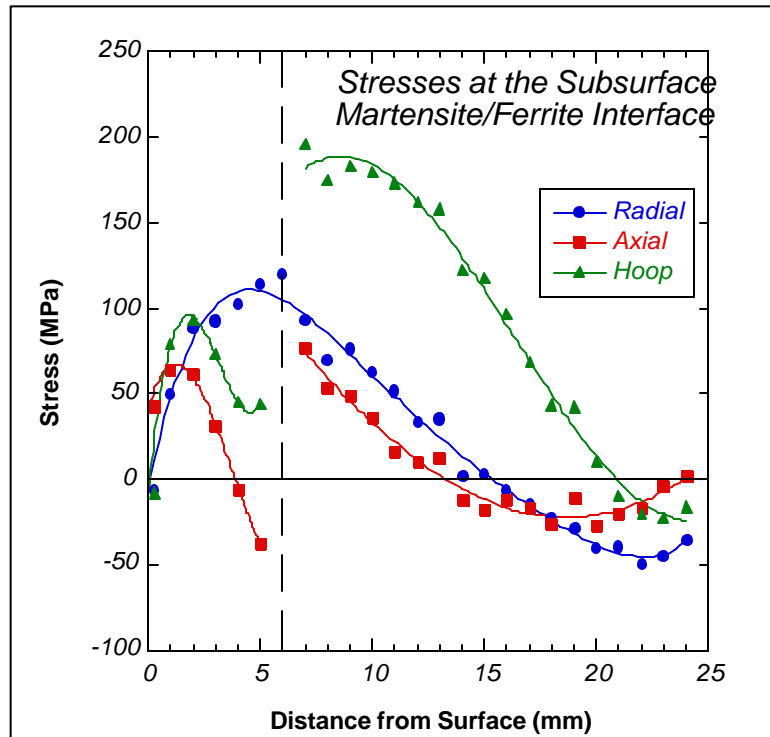
Collaboration amongst:

HTML – Ohio State University - Honda Engine (Marysville, OH)

Residual stress: Neutron and x-ray diffraction techniques employed



Induction hardened steels - a cost saving process



- Induction hardened crankshafts
 - More reliable
 - Lower cost via energy savings
- Renewed interest in process
- Residual stress: Neutron and x-ray diffraction techniques used
- EERE State Partnership Program:
 - HTML@ ORNL
 - Cummins Engine
 - Indiana State Energy Office



Some FY 2001 Projects Have Been Especially Noteworthy: Machining/Tribology

- **Wear of EDM Recast Layer (NCSU)**
- **Grinding wheel and coolant studies (Milacron)**
- **Development of standard testing procedure (ABC/Techsolve/Milacron)**
- **Tribology of advanced nickel based coating (US Chrome)**
- **NDE detection of grinding flaws (Cummins/INCO/Coorstek)**

Low-Cost Manufacturing of Precision Diesel Engine Components



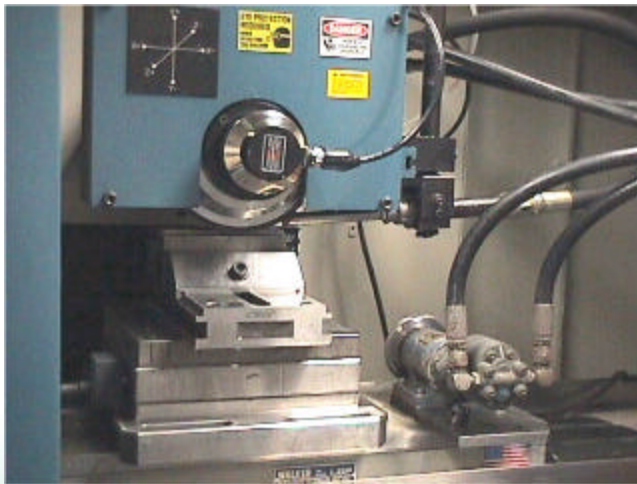
Instrumented Weldon Cylindrical Grinder



Ernst Leitz Scanning Acoustic Microscope

- **Objectives**
 - Cost-effective fabrication of precision diesel engine components
 - Cost-effective, non-destructive methods for detecting and preventing machining-induced damage in engine components.
- **Benefits**
 - Improved engine durability, performance and fuel economy
- **Recent Progress**
 - Scanning Acoustic Microscope used to detect sub-surface cracks in ceramic components produced by overly aggressive grinding.

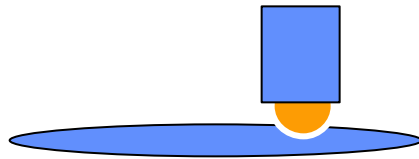
Superabrasive Grinding Wheel Performance Evaluation



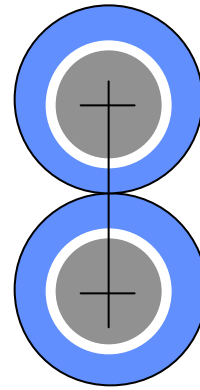
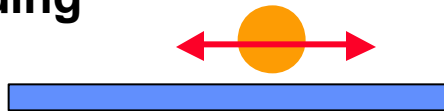
- **Objective**
 - Standardized methods for evaluating and comparing the performance of superabrasive grinding wheels
- **Benefits**
 - Improved wheel performance
 - Reduced production cost for ground engine components
- **Recent Participants at the HTML**
 - American Boarts Crushing Company
 - Saint Gobain Superabrasives
 - Milacron Abrasive Products

A Variety of Friction and Wear Testing Geometries is Available

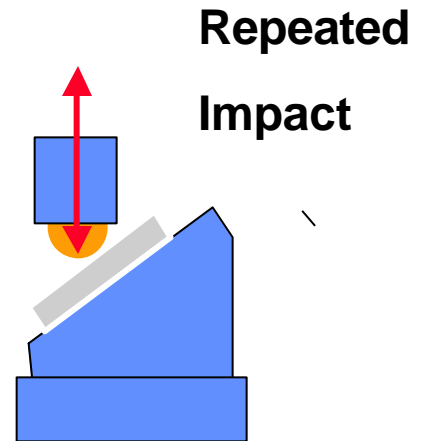
Pin-on-disk



Reciprocating sliding

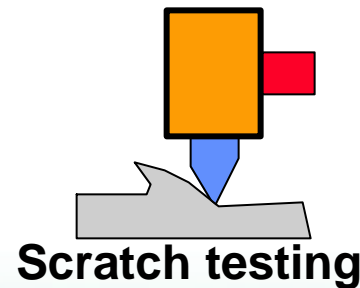


Disk-on-disk

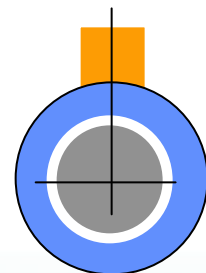


Block-on-ring

- Room or high temperature
- Custom tests / simulations
- Lubricated or dry
- Controlled atmosphere



Scratch testing



HTML User and Fellowship Programs Have Hosted Numerous Diesel Projects

- **CATERPILLAR: User Projects**
 - **Characterization of VLS Silicon Nitride Fibrils**
 - **Residual Stress Determination of TTBC Systems**
 - **Effect of Interference Fit on Residual Stress of Zirconia/Gray Iron System**
 - **Residual Stress Measurement for Characterizing Material Response to Arc Welding**

Fellowship Projects

- **Jian Zhang: Characterization of Thin Film Coating Properties for Heavy Equipment Applications**
- **Cynthia Hsieh: Characterization of Submicron Particles for Developing Innovative Steels for Heavy Equipment Applications**

HTML User and Fellowship Programs Have Hosted Numerous Diesel Projects

- **DDC: User Projects**
 - **Identification of Typical Microstructures and Phases of Sintered Silicon Nitride Cam Roller Followers**
 - **Effects of Manufacturing and Service Parameters on Properties of a Heat Engine Component**
 - **Cost-Effective Ceramic Engine Components**
 - **SIALON Valves Investigation**

HTML User and Fellowship Programs Have Hosted Numerous Diesel Projects

- **CUMMINS: User Projects**
 - **Studies of Environmentally Induced Fracture of Diesel Engine Cylinder Head Cap Screws**
 - **Residual Stress Measurements in Thermal Barrier Coatings**
 - **Microstructural Characterization of Electrochemical Cells**
 - **Microstructural Characterization of Oxidation Catalysts**
 - **Characterization of Oil Balls and Their Influence on Premature Overlay Removal of Diesel Engine Bearings**
- **EATON:**
 - **Development of Techniques for the Analysis and Characterization of RBSN and SRBSN**
- **NORTON:**
 - **Mechanical Characterization and Analysis of Ceramic Valves for Diesel Engines**

HTML User and Fellowship Programs Have Hosted Numerous Automotive Projects

- **Ford Motor Company (Ford Research Lab):**
 - Residual Stress Analyses on Al319 Thermally Sprayed Coatings
 - Effect of Thermal Cycling on the Thermal Conduction of HCC-MMC's (High Ceramic Content Metal Matrix Composites)
 - Influence of Brake Rotor Design and Heat treatment of residual Stress of Grey Cast Iron Rotors
 - High Resolution Microscopic Study of Copper-MFI Catalysts
- **General Motors (North American Operations):**
 - TEM Investigation of Precipitation in Metal Matrix Composites
- **Delphi**
 - Thermal Properties of Magnaquench

HTML User and Fellowship Programs Have Hosted Numerous Automotive Projects

- Catalysts (Partial Listing):**

CUMMINS- Microstructural Characterization of Oxidation Catalysts

ENGLEHARD- Thermal Cycling Behavior of Pd-PdO on Alumina

FORD- High Resolution Microscopic Study of Copper-MFI Catalysts

W.R. GRACE- Characterization of Deactivation Mechanisms in a
Volatile Organic Carbon Catalyst System

UNITED EMISSION CATALYSTS- Optimization of the Activity of
Manganese-Based Oxidative Catalysts

UN. DELAWARE- HRTEM of Mo-Promoted Rh on Gamma Alumina

UN. DELAWARE- HRTEM Study of Microstructure Development in
Polyfurfural Alcohol Derived Carbogenic Molecular Sieves

UN. KENTUCKY- Crystal Structure Identification of Pt-Re-Al₂O₃
Catalysts Using HTXRD

UN. KENTUCKY- Crystal Phases in Pt-Sn Catalysts

UN. NEW MEXICO- Electron Holography of Catalytic Materials

The HTML:

**It's where you go to get it
done!**